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Selection of 3013 Containers for Field Surveillance: Fiscal Year 2021 Update

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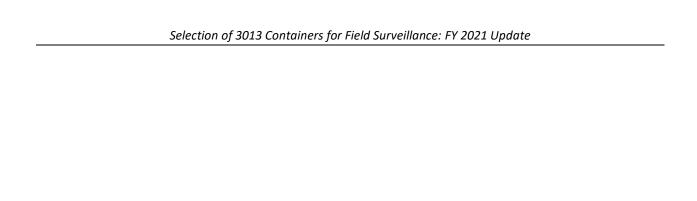
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ACRONYMS

Am americium

ARIES Advanced Recovery and Integrated Extraction System

BDT binning decision tree

C&D Cats and Dogs (containers)

Cl chloride

CWG Corrsoion Working Group
DE destructive examination

DFF Dynamic Flowform
DMO Direct Metal Oxidation
DOE Department of Energy
EJ engineering judgment

ER engineering review

F fluorine

FTIR Fourier transform infrared (spectroscopy)

FY fiscal year

HCl hydrogen chloride

ICCWR inner container closure weld regionISP Integrated Surveillance ProgramLANL Los Alamos National Laboratory

LCM laser confocal microscope

LLNL Lawrence Livermore National Laboratory

LOI loss on ignition

MIS Materials Identification and Surveillance

MOX Mixed Oxide

MS mass spectroscopy

NDE nondestructive examination

Np neptunium

P&C pressure and corrosion

PCD Product Certification Database

PG prompt gamma ppm parts per million

Pu plutonium

RFETS Rocky Flats Environmental Technology Site

SCC stress corrosion cracking

SME subject matter expert

SPD surplus plutonium disposition

SRS Savannah River Site

TGA thermal/thermogravimetric analysis

U uranium

WAMS Wide-Area 3D Measurement System

WG Working Group wt% weight percent

XRT X-Ray Tomography

ABSTRACT

This Update is the ninth in a series of reports that document the binning and sample selection of 3013 containers for the Field Surveillance program as part of the Integrated Surveillance Program. The last Update was in 2016. This Update documents changes to the binning and changes to the random and engineering judgment samples since 2016. This Update also documents field surveillance activities since 2016 and describes plans to complete random sampling in 2025. In addition, this Update describes a new effort to collect surveillance data as part of down blending operations and updates assumptions about Advanced Recovery and Integrated Extraction System (ARIES) 3013 containers.

1.0 Introduction

A Department of Energy (DOE) standard, "Stabilization, Packaging, and Storage of Plutonium-Bearing Materials" (DOE-STD-3013) (DOE 2018), was issued to define the stabilization and packaging requirements that assure excess plutonium can be safely stored for up to 50 years. Packaging of plutonium bearing materials into 3013 containers began in 2001. The most current guidance for the Integrated Surveillance Program (ISP) is described in "Integrated Surveillance and Monitoring Program for Materials Packaged to Meet DOE-STD-3013" (DOE 2015). This 2015 ISP document replaces the original ISP described in Surveillance and Monitoring Plan for DOE-STD-3013 Materials" (DOE 2003). As of July 2021, there are 4,728 3013 containers in the ISP database remaining in the inventory. The majority of these are stored at Savannah River Site (SRS). Some are temporarily staged at Pantex, Nevada National Security Site, Idaho National Laboratory and Los Alamos National Laboratory (LANL). The number of containers is reduced from the number in the 2016 update due to destructive examinations, down blending and other material disposition activities.

A 3013 container can be certified if the packaging facility certifies that all of the requirements in the 3013 Standard are met. However, that does not mean it is part of the ISP. To be part of the ISP, the container must be compliant with the Standard and all required data must be in the ISP database (DOE, 2015). If the container is to be shipped to SRS, then it must also meet the applicable requirements of the 20 Points document (SRS, 2000) to assure that the container can be safely stored in K-Area in compliance with their Documented Safety Analysis

The 2015 ISP document summarizes surveillance findings and Shelf-life testing results, concludes that pressurization is no longer considered a potential container failure mechanism, and directs that the ISP be restructured to focus on the potential for stress corrosion cracking (SCC) of the containers. It draws extensively from the "Test Plan for Assessing Potential for Stress Corrosion Cracking in the 3013 Inner Container Closure Weld Region (Fiscal Year [FY] 2014)" (Berg, et. al 2014).

The ISP is a combination of two focused activities, Field Surveillance and Shelf-life testing, to ensure the safe long-term storage of the 3013 containers. Field Surveillance program staff

examine containers randomly selected from the storage inventory and containers selected based on the Materials Identification and Surveillance (MIS) Working Group's (WG) engineering judgment (EJ) that the containers are most likely to exhibit degradation based on their known attributes (e.g., moisture content, chloride content, etc.). The MIS-WG is comprised of a subject matter expert (SME) from each of the DOE sites packaging or storing certified 3013 containers. Shelf-life testing includes representative and other materials tested in an accelerated manner to evaluate potential degradation mechanisms. Plutonium stabilization and packaging into 3013 containers began in 2001, Shelf-life testing began in 2001 and Field Surveillance started in 2005.

This Update is the ninth in a series of documents that describe and provide guidance for the Field Surveillance program. Updates have been issued as additional containers are generated, additional information has been obtained on containers, and to incorporate results from Field Surveillance and Shelf-life testing. In 2005, three reports were published documenting the binning approach "Binning of 3013 Containers for Field Surveillance" (Peppers et al., 2005a), the sampling approach "3013 Surveillance Sampling—The Statistical Sample" (Kelly et al., 2005), and the items in the statistical (random) and judgmental samples "3013 Container Statistical and Judgmental Samples Selected for Non Destructive Evaluation (NDE) in FY 2005" (Peppers et al., 2005b). In 2007, these three reports were combined into one document, "Selection of 3013 Containers for Field Surveillance" (Peppers et al., 2007), and the binning and sampling information was updated. Field Surveillance Destructive Examinations (DE) began in 2007. In 2009, "Selection of 3013 Containers for Field Surveillance, Revision 1" (Peppers et al., 2009) was published. Readers unfamiliar with the 3013 Field Surveillance program are encouraged to read Peppers et al. (2009) for a thorough historical perspective.

In 2011, "Selection of 3013 Containers for Field Surveillance: 2011 Update" (Kelly et al., 2011) provided an update to the comprehensive Peppers et al. report (2009). In 2013, "Selection of 3013 Containers for Field Surveillance: 2013 Update" (Kelly et al., 2013) updated the information in Kelly et al. (2011). In 2016, "Selection of 3013 Containers for Field Surveillance: 2016 Update" (Kelly et al., 2016) documented the random and EJ DE items in FYs 2013, 2014, and 2015. (Note that DE surveillances are identified by the budgetary fiscal year in which they are performed.)

The 2016 Update describes the restructuring of the Field Surveillance program as defined by the 2015 ISP document for FY 2016 and beyond. This restructuring focused DEs on Pressure and Corrosion (P&C) items with best moisture levels greater than or equal to 0.08 wt%. See Section 3.2 for a discussion of best moisture versus certified moisture levels. The Shelf-life studies and surveillance findings behind this restructuring are described in DOE 2015. In the 2016 Update, it was determined that a minimum of six DE containers (random plus EJ) per year was appropriate. However, in FY 2019 only one container was examined. This resulted in an increase from six to seven containers per year in FY 2020 and beyond to maintain the random sampling completion date of 2025.

This Update, "Selection of 3013 Containers for Field Surveillance: 2021 Update," documents the DEs performed in FY 2017 through FY 2021. It also documents the proposed items for FY 2022

(Table 5-1) and provides a list of potential future EJ DEs (Table 5-2). Appendix A contains a list of the remaining higher moisture S₁ (see Section 2.0) P&C containers, which includes the fifteen random sample containers that will remain after FY 2022. In addition, this Update describes the protocol for DE inner container closure weld region (ICCWR) examinations (Section 6) and the protocol for collecting surveillance inspection information during down blending operations at SRS (Section 7). It also updates assumptions about the packaging of future ARIES 3013s (Section 8).

2.0 BACKGROUND FOR BINNING AND SURVEILLANCE SAMPLE SELECTION

As described in DOE 2015 and the previous guidance (LANL, 2001 and DOE, 2003), two potential mechanisms for container failure were identified: over-pressurization and corrosive degradation of the 3013 container. The container inventory was sorted into three bins based on the potential for experiencing the identified degradation mechanisms: the Innocuous bin (pressurization and corrosion unlikely), the Pressure bin (pressurization possible, corrosion unlikely), and the Pressure and Corrosion (P&C) bin (both pressurization and corrosion possible).

For the Pressure bin and the P&C bin, the random sample selection was based on the criterion of achieving a 99.9% probability of examining at least one of the worst 5% of the containers in that bin. This requirement is referred to as the 99.9% / 5% confidence criterion. To meet this requirement 130 containers were randomly selected from the Pressure bin and 128 containers were randomly selected from the P&C bin.

Through FY 2014, NDE was performed on 152 containers (140 random and 12 EJ). The 140 randomly selected containers included the 130 Pressure bin containers and 10 Innocuous bin containers. None of the NDE containers showed pressurization (Yerger et al, 2010). In addition, destructive examination (DE) was performed on 93 containers (63 random and 30 EJ) from the three bins combined. The maximum pressure observed during DE of 3013 Standard compliant containers was 21 psia, well below the 714 psia (699 psig) working pressure of the outer container. One container that was packaged with 0.53 wt% moisture (H003328), thus not meeting the 3013 packaging requirements, was opened at LANL. Using equivalent equipment and calculation methodology, the pressure and gas composition were measured. Even this container had a pressure less than 29.3 psig. Based on these findings and results from Shelf-life testing, the MIS-WG has concluded that it is unlikely that pressure will be a failure mechanism for 3013 containers (DOE, 2015). Therefore, examination of random containers selected from the Pressure and Innocuous bins has been completed (DOE, 2015).

However, results show that corrosion is still a potential mechanism for container degradation — especially for containers in the P&C bin that have elevated (but still 3013 Standard compliant) moisture content. Although no container failures have been detected, pitting corrosion and small crack features have been observed in some containers during DE and it is possible for similar pits to develop into stress corrosion cracks over time (Berg, et. al 2014).

Based on the recommendation of the MIS-WG, the Surveillance and Monitoring Program was restructured in 2016 to focus on the potential for stress corrosion cracking in the ICCWR of the 3013 containers (Kelly et al., 2016). The restructuring focused DE examinations on the higher moisture containers (>= 0.08 wt%). This population is denoted as S_1 and the containers with moisture less than 0.08 wt% are denoted as S_2 . The Shelf-life studies and surveillance findings behind this restructuring are described in DOE 2015 and Kelly et al. 2016. Based on expert judgment, the S_1 population is assumed to be at least five times more likely to have a worst case container than the S_2 population (Kelly et al., 2016).

The confidence criterion for the surveillance sample remains the same, achieving a 99.9% probability of examining at least one of the worst 5% of the containers in the P&C bin. Since worst is now focused on the potential for SCC in the ICCWR, the sample selection is now focused on the S_1 population.

3.0 BINNING

Binning is a key component of the statistical sampling approach. Although the emphasis for future field surveillance DEs is on the P&C bin, historically, binning consists of a two-tiered review of all 3013 containers with the primary objective of placing each container into one of the three bins (Innocuous, Pressure, or P&C) for the purpose of surveillance.

3.1 Binning Decision Flow

The binning decision flow (Figure 3-1) has two tiers as described below.

Tier 1—Decision Tree Up to Engineering Review (ER): containers that have already been packaged are assigned to the appropriate surveillance bin based on information in their data packages.

Tier 2—ER: containers that have already been packaged but fall through the initial decision tree screening require an ER before they are assigned to an appropriate bin.

Information to facilitate binning of existing containers comes from the ISP database. The ISP database has several modules. The module used for binning is the Product Certification Database (PCD). It contains all of the information generated by the packaging sites, as well as additional data from reevaluation of existing data present in the database (e.g., moisture data). The PCD includes information such as MIS Represented group designation (referred to as the 3013 taxon) (Narlesky et al., 2009), moisture content of the material, prompt gamma (PG) analytical data taken after packaging, and chemical analysis data when available (Friday et al., 2010).

Initial Binning of Materials. The initial binning evaluation has six principal binning tree decision (BDT) points (BDT-1 through BDT-6 in Figure 3-1). These are evaluated using a Microsoft Access SQL (structured query language) macro applied to the PCD. This was first documented in Appendix A in Kelly et al. 2011 and updated in Appendix B of Kelly et al. 2016.

The first decision point assigns materials consisting of plutonium metal and associated impurities to the Innocuous bin (BDT-1). The second, third, and fourth decision points identify containers having the potential for corrosion. The primary constituent for causing corrosion is chloride salts or possibly fluoride-containing materials. Using information from the database, containers identified as containing either chlorine (Cl) or fluorine (F) are placed in the P&C bin. Identification of chlorine or fluorine can be accomplished by chemical analysis (BDT-2), PG analysis (BDT-3) or process knowledge of the material (BDT-4). Chemical data are limited to a small number of Hanford containers and based on results reported by Tingey and Jones (2005).

These methods for determining the presence of corrosive materials have varying degrees of accuracy and sensitivity. For example, if the chemical analysis shows Cl greater than 1,000 ppm or F greater than 8,000 ppm (BDT-2) or if the PG analysis detects either Cl (any positive detection) or F greater than or equal to 0.8 total weight percent (wt%) (8,000 ppm) (BDT-3), the container is placed in the P&C bin. The PG detection limit for Cl is about 0.8 wt%, and the detection limit for F is about 0.1 wt%. The threshold of concern for Cl is below the detection limit of PG, so if the material in the container originated from a process that may have introduced chlorides, it is placed in the P&C bin (BDT-4), unless there is additional analytical information to the contrary. The inset box from BDF-4 in Figure 3-1 contains a list of the 3013 represented groups (taxons) that were assigned to the P&C bin based on process knowledge (BDT-4).

The MIS Represented Group (3013 taxon) is a designation given to each packaged 3013 container in the ISP database and each characterized item in the MIS Module. The purpose of the 3013 taxon is to match each 3013 container to the representative Shelf-life data of the MIS items. The 3013 taxons are assigned based on process knowledge, which links 3013 containers and MIS items produced by similar processes, or item-by-item linkages, which links 3013 containers and MIS items based on PG analysis, and/or by a thorough review documented in a report (e.g. 3013 containers represented by MIS Item 011589A) (Narlesky et al., 2009).

The fifth decision point is based on the final moisture content of the oxide (BDT-5). The DOE-STD-3013 (DOE, 2018) sets the moisture limit for oxide materials at 0.5 wt%. However, the actual acceptance limit for moisture content varies depending on the method for moisture analysis and the uncertainties and biases associated with the particular method. For oxide materials without corrosive species (i.e., materials that pass the Cl and F screen), containers with a loss on ignition (LOI) result greater than or equal to 0.05 wt% are assigned to the Pressure bin. Containers with thermogravametric analysis (TGA), TGA with Fourier transform infrared (TGA-FTIR or FTIR), or TGA with mass spectroscopy (TGA-MS or MS) moisture measurements greater than or equal to 0.10 wt% are assigned to the Pressure bin. If there is no PG measurement, the container must have an ER (see side box on Figure 3-1).

The sixth (BDT-6) and last decision point of the initial binning protocol is based on the combined wt% of plutonium (Pu), americium (Am), and neptunium (Np). Containers in which this combined total weight percent is greater than or equal to 85% are placed in the Innocuous bin unless the fluoride or PG exception applies. Uranium (U) is excluded from the initial binning

process because its large measurement uncertainty could skew the binning results. Note that these materials are considered for these purposes to be pure oxide with moisture content below the specified limits. If a container successfully passes the screening test for P&C as well as for Pressure, and had less than 85 wt% Pu + Am + Np, it requires an ER by a committee of experts.

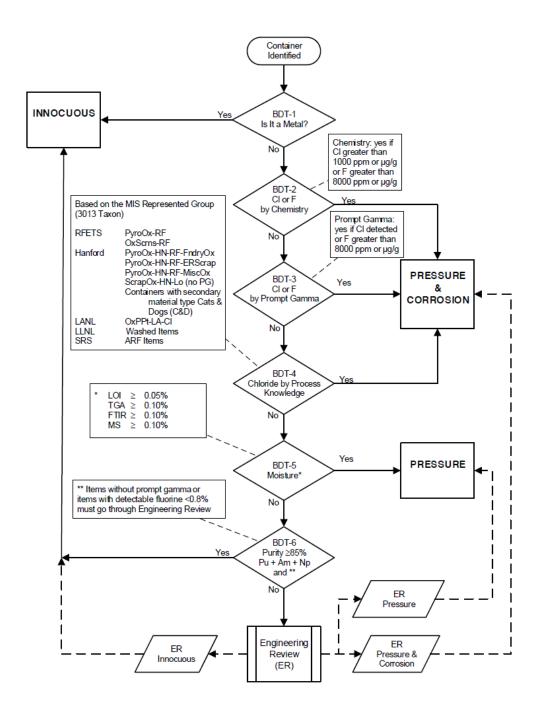


Figure 3-1. Generic decision tree for binning 3013-type containers for Field Surveillance

Binning by Engineering Review. Packaged material that is not assigned a bin using the initial binning protocol described above is required to undergo an ER. All packaged containers subject to ER have a Pu + Am + Np content of less than 85 wt% (or meet the detectable low fluoride or without PG exception) with no known chloride content from process knowledge or analytical analyses and have a moisture content of less than 0.05 wt% by LOI or less than 0.1 wt% by TGA and/or FTIR/MS. The presence of U is addressed during the ER.

In addition, if an anomaly arises such that the container situation is not covered in the binning decision flow, it will have an ER. This situation was only recently encountered when a Lawrence Livermore National Laboratory (LLNL) container did not have a moisture measurement (Riley, 2020).

The criteria that are used in the ER are described below.

- **Criterion 1**: Containers with greater than 85 wt% Pu + Am + Np + U (total actinide) that have PG are placed in the Innocuous bin. These containers are reviewed on an individual basis to ensure that the material comes from a historically pure stream so that the uranium measurement uncertainty cannot cause an impure material to be binned as innocuous. Containers without PG require further review and can be assigned to any of the three bins.
- **Criterion 2**: Containers with total actinide content between 80 and 85 wt% are reviewed on an individual basis. Those containers from a process that historically produced pure material and have a measured moisture content <0.05 wt% are placed in the Innocuous bin unless there is a suspected problem with the moisture analysis identified through a nonconformance report or other documented production comment. Containers not meeting the moisture criteria are placed in the Pressure bin.

The only exception to the moisture criterion is for mixed plutonium-uranium oxide containers processed in the stabilization packaging equipment dry line at Hanford. If these containers have a measured moisture value exceeding 0.05 wt%, the results are reviewed on an individual basis to determine if excess weight loss occurred at high temperatures and can be attributed to oxygen loss from the uranium oxide and not water. For these cases the container is placed in the Innocuous bin.

- **Criterion 3**: Containers with a total actinide content of less than 80 wt% are placed in the Pressure bin. (Exceptions are oxide containers evaluated under Criterion 4.)
- **Criterion 4**: Oxide containers produced by magnesium hydroxide precipitation from pure plutonium nitrate solutions represent a special class of items where the major impurity is magnesium oxide and PG indicates no other significant impurities.

<u>Hanford</u>—Containers from Hanford packaged in the stabilization packaging equipment dry line and having measured moisture content of less than 0.05 wt% are placed in the Innocuous bin. All others are placed in the Pressure bin.

<u>RFETS</u>—Containers from Rocky Flats Environmental Technology Site (RFETS) must have a measured moisture value of less than 0.05 wt% and the glovebox moisture content at the time of packaging must be less than 1,000 ppm. Containers meeting these criteria are placed in the Innocuous bin. All others are placed in the Pressure bin. Containers suspected to have originated from other than pure plutonium nitrate (e.g., Pu/U solutions) are evaluated using Criteria 1, 2, or 3.

Criterion 5: This criterion applies only to RFETS containers; similar data are not available from other sites. During the moisture analysis using TGA-FTIR, evaluation of the FTIR data indicated the evolution of hydrogen chloride (HCl) from some samples (Berg et al., 2005). HCl was found to occur in three temperature ranges: 20°C–350°C, 350°C–670°C and 670°C–1,000°C. However, only the HCl values in the low temperature range are important to the material storage temperatures because the material temperatures are not expected to exceed 350°C. A total of 36 containers with low temperature HCl have been found in the RFETS inventory with four of those containers in the ER category. This analytical method is very sensitive and possibly subject to contamination from other chloride-bearing samples. However, taking a very conservative approach, all 36 containers are placed in the P&C bin. It is probable that other sites have materials that could exhibit this property, but these cannot be evaluated and are left in their assigned bins.

Sub-bins. Each 3013 container that was assigned a primary bin using the protocol described above was also given a secondary hierarchical classification or sub-bin. The sub-bin provides additional detail that identifies the criterion that was used to assign the primary bin (Table 3-1). For example, BDT-4-SR-ARF means that the container was placed in the P&C bin because of process knowledge as defined in decision point number four. The sub-bin ER-C2-P means that the container was placed in the pressure bin based on an ER and that the material's total actinide content ranged between 80 and 85 % wt (criterion 2). Any sub-bin of the form ER-xx-E-x refers to an "exception" associated with unique properties as determined by an ER. A total of 35 distinct sub-bin designations were used in the binning assignments.

3.2 Binning Changes Resulting from Best Available Moisture Measurements Determined after FY 2016 Update

When 3013 containers are loaded, the packaging sites are required to certify that the moisture content, including measurement uncertainty, is below the 3013 Standard limit of 0.5 wt%. The packaging sites use one of several approved moisture measurement techniques. Some of these techniques overstate the moisture content to varying degrees. For example, TGA to 1,000°C will report all of the mass lost during heating (water, carbon dioxide, volatilized salt, etc.) as water. This is conservative when assuring compliance with the 3013 Standard, but it can be somewhat misleading when evaluating corrosion as a function of moisture content. The LOI technique can either over report water (due to loss of volatile content other than water) or under report water (due to readsorption of water from the glovebox atmosphere after cool down and before making the final mass measurement). Because of the potential for under reporting, when the LOI technique was used the packaging facility imposed a lower acceptance criterion (generally in the

0.2 wt% range) to bound the amount of water that could be readsorbed. In addition, the use of LOI was restricted to plutonium oxide with minimal impurities.

Table 3-1. Sub-bin Designations and Definitions

FY 2011 SubBin	Basis for Binning/Sub-Binning Determination
BDT-1-I	Physical form of material was metal (decision point 1)
BDT-2-Cl	Chemical data for chloride (decision point 2)
BDT-2-F	Chemical data for fluoride (decision point 2)
BDT-3-Cl	Prompt gamma data for chloride (decision point 3)
BDT-3-F	Prompt gamma data for fluoride (decision point 3)
BDT-4-H-1E	3013 taxon was Hanford 1E (i.e., PyroOx-HN-RF-ERScrap, PyroOx-HN-RF-FndryOx, or PyroOx-HN-RF-MiscOx)
BDT-4-H-2B	3013 taxon was Hanford 2B (ScrapOx-HN-Lo and no prompt gamma was performed)
BDT-4-H-CD	Hanford Secondary Material Type C&D
BDT-4-LANL-Cl	LANL oxalate precipitation-aqueous chloride (decision point 4)
BDT-4-LLNL- WASHED	Lawrence Livermore National Laboratory (LLNL) washed items (decision point 4)
BDT-4-RF-2B	3013 taxon was RFETS ¹ 2B (PyroOx-RF)(decision point 4)
BDT-4-SR-ARF	Savannah River ARF items (decision point 4)
BDT-5	Binning was based on moisture (decision point 5)
BDT-6	Binning was based on total weight (%) of Am, Np, and Pu (decision point 6)
ER-BDT-6-I (Low F)	ER because of low fluoride (decision point 6)
ER-BDT-6-I (No PG)	ER because no prompt gamma (PG) was performed (decision point 6)
ER-C1-I	ER based on the total mass (%) of Am, Np, Pu, and U
ER-C1-I (No PG)	ER because no PG but was performed
ER-C1-P	ER (criteria 1) - pressure bin
ER-C1-P (Low F)	ER (criteria 1) - pressure bin (low fluoride content)
ER-C1-P (No PG)	ER (criteria 1) -pressure bin (no PG)
ER-C2-E-I	ER (criteria 2) - designated as an exception
ER-C2-E-P	ER (criteria 2) - designated as an exception
ER-C2-I	ER (criteria 2) - innocuous bin
ER-C2-I (Low F)	ER (criteria 2) - innocuous bin (low fluoride content)
ER-C2-I (No PG)	ER (criteria 2) - innocuous bin (no PG)
ER-C2-P	ER (criteria 2) - pressure bin
ER-C2-P (Low F)	ER (criteria 2) - pressure bin (low fluoride content)
ER-C2-P (No PG)	ER (criteria 2) - pressure bin (no PG)
ER-C3	ER (criteria 3)
ER-C3 (Low F)	ER (criteria 3) - low fluoride content
ER-C3 (No PG)	ER (criteria 3) - no PG
ER-C3-E-P	ER (criteria 3) - but designated as an exception
ER-C3-P	ER (criteria 3) - pressure bin
ER-C4-I	ER (criteria 4) - innocuous bin

For surveillance recommendations in this revision, we use the most accurate available moisture measurements rather than measurements designated to certify the moisture content at the time of packaging. The is referred to as the "best moisture" measurement for a container and is defined

as that measurement best reflecting the true moisture content from among the available data collected at the time of packaging. For the TGA example, the certified moisture in the PCD may be the TGA result, but in some cases MS or FTIR results may also be available. MS and FTIR are direct measurements of water driven off of the sample during the TGA. If only TGA results are available, the mass loss to 650°C is more representative of the actual moisture content, since it does not include the mass loss from salt volatilization. For containers that used LOI, no better estimate is possible, so the LOI value is used.

The best moisture is the moisture value obtained by the highest ranked measurement method that was employed for that sample, with the ranking shown in the following list (highest to lowest) of methods applied for each packaging site. The list matches the possible values found in the "MoistureMethod" field of ISP database:

```
RFETS
      FTIR Recalculated*
      FTIR
      TGA
      LOI
Hanford
      TGA-MS w StorWtGain
      TGA-MS, no StorWtGain
      AvgOfTGAto650plusStorWtGain*
      TGA w StorWtGain
      TGA, no StorWtGain
      LOI
SRS
      MS
      AvgOfTGAto650plusStorWtGain*
LANL
      TGA-MS (or TGA/MS)
      LOI
LLNL
      LOI, Full Batch
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Although changes in moisture measurements do not affect which items go into the P&C bin, they do affect the number of items in the P&C bin determined to have moisture levels greater than or equal to 0.08 wt%. A revised best moisture analysis after the 2016 Update resulted in 26 RFETS items having moisture levels less than 0.08% that were previously identified as greater than 0.08 wt%. As a result of this change, the required sample size for the 99.9% / 5% confidence requirement went from 62 to 61 S₁ containers. This calculation is based on containers in the PCD and an evaluation of the 3013 containers packaged and stored at LANL. This analysis showed that there are currently 11 P&C containers stored at LANL. Of these 11, four have LOI moisture measurements greater than 0.08 wt% (Kelly et al., 2016). Of the 26 RFETS items moving to S₂ only one was in the random sample, R610832. It was removed from the sample leaving the required 61 previously identified in 2016.

^{*} The MoistureMethod values marked by an asterisk were added to the ISP database after items had been placed in storage by reanalyzing data collected at the time of packaging.

4.0 FIELD SURVEILLANCE SAMPLING FROM FY 2017 TO FY 2021

As stated in Section 3.2, to meet the confidence criterion (99.9% / 5%), sixty-one DEs randomly selected from S_1 are needed to evaluate corrosion in the ICCWR. Table 4-1 shows all the containers that have undergone destructive evaluation since 2013. In the table random containers are indicated by R and engineering judgement containers are indicated by J. The containers with best moisture greater than 0.08 wt% are indicated with S_1 and with less than 0.08 wt% with S_2 .

Table 4-1. All Containers with DEs Since FY 2013

DE	Container ID	J or R S1 or S2	DE	Container ID	J or R S1 or S2
13DE01	H001236	J/S1	17DE02	H002575	R/S1
14DE01	R610996	J/S2	17DE03	H003352	R/S1
14DE02	H003064	R/S1	17DE04	H003695	R/S1
14DE03	H003307	R/S1	17DE05	H002508	R/S1
14DE04	H003052	R/S1	17DE06	R600793	J/S1
14DE05	H003898	R/S1	18DE01	H003345	R/S1
14DE06	S002277	J/S1	18DE02	H003626	R/S1
14DE07	S002116	R/S1	18DE03	H003645	R/S1
14DE08	H004219	R/S1	18DE04	H002524	R/S1
14DE09	H002636	J/S2	18DE05	H003523	R/S1
15DE01	R610156	R/S1	18DE06	H004153	J/S1
15DE02	S002162	R/S1	19DE01	A000632	J/Pressure
15DE03	H001979	R/S1	20DE01	H003308	R/S1
15DE04	H001181	J/S1	20DE02	H003311	J/S1
15DE05	H003181	R/S1	20DE03	H003676	J/S1
15DE06	H003258	R/S1	20DE04	H004005	R/S1
15DE07	H003737	J/S1	20DE05	H002531	R/S1
15DE08	H003896	R/S1	20DE06	H004226	J/S1
15DE09	H004302	R/S1	20DE07	H003271	J/S1
16DE01	H001191	J/S1	21DE01	H004216	J/S1
16DE02	H002556	R/S1	21DE02	H003731	R/S1
16DE03	H004173	R/S1	21DE03	S002151	R/S1
16DE04	H004247	R/S1	21DE04	R610910	R/S1
16DE05	H003775	R/S1	21DE05	S002219	R/S1
16DE06	H004024	R/S1	21DE06	H001746	R/S1
17DE01	H001304	R/S1	21DE07	H003564	R/S1

There are 41 S₁ items credited as being in the random sample that have been DE'd as of the end of FY 2021 (highlighted in Table 4-1). Included in the 41 random sample containers are 17 S₁ containers that were DE'd before 2016 and have lids available for ICCWR examination. Although four of these were selected by EJ, MIS has decided to keep all 17 as part of the random sample. This decision could slightly increase a bias towards potential problem containers. Such a bias is not considered problematic, since it is likely to be small and, if it exists at all, will be conservative.

In FY 2016 five randomly selected S₁ containers were DE'd (Table 4-1). In FY 2017 and FY 2018, six S₁ containers were DE'd each year and of these five were randomly selected for a total of 10 additional randomly selected S₁ containers with lids available for ICCWR examination. Only one container was DE'd in FY19, it was a LANL EJ item that was in the Pressure bin. In FY 2020, seven containers were DE'd, and of these three were randomly selected. In FY 2021 seven containers were examined and of these six were randomly selected.

Twenty more random sample containers are needed to complete the 99.9% / 5% confidence criterion. Five random items and two EJ items per year are currently planned in FY 2022 – FY 2025. This means that 61 S₁ random sample containers (including the 4 EJ discussed above) will have been DE'd, in the sense of opened and their lids made available for ICCWR examinations, by the end of FY 2025. In addition, from 2016 through 2025, 16 S₁ EJ containers will have been DE'd, again in the sense of opened and lids made available for examination. This information is summarized in Table 4-2.

Table 4-2. Summary of Random Sample and EJ Containers Planned for DE by the End of FY 2025

Number of DE Random Sample Containers with ICCWR Examinations (Number of EJ)	Year
17 (4 EJs included in random) (0)	pre-2016
5 (1)	2016
5 (1)	2017
5 (1)	2018
3 (4)	2020
6 (1)	2021
5 (2)	2022
5 (2)	2023
5 (2)	2024
5 (2)	2025
61 (16)	End of FY 2025

5.0 Planned Field Surveillance Sampling from 2022 Through 2025

Table 5-1 shows the proposed seven containers, two EJ and five random, selected for field surveillance in 2022.

Selection Site **ISP Bin** 3013 Container ID **Surveillance Comment** (Packaged) Type High best moisture with H004179 Engineering Pressure and Corrosion Hanford additional selection criteria Judgment documented in the DE letter H003697 H003715 Hanford Items in the Random Sample with H004183 Hanford Pressure and highest best moisture with Random H004006 Hanford constraints documented in the DE Corrosion letter H003945 Hanford H004004 Hanford FY 2022 Total 7

Table 5-1. Selection of FY 2022 3013 DE Surveillance Samples

Container H004179 was selected for examination because of its primary material type (S - sweepings), which is different than most of the others (ARF) from Hanford that have been examined. In addition, it has Cl at 8.6 wt%, moisture at 0.25 wt% and a weight gain of 0.9 g. The other FY 2022 EJ item, H003697, was selected because in addition to high moisture, it had to be re-stabilized prior to packaging.

The MIS Working Group also identified twenty S₁ containers that are of interest for future examination based on engineering judgment. Table 5-2 shows these containers and the reason for their selection. ¹

The intent of identifying future EJ 3013s is to prevent items from going to down blending prior to DE, get them removed from the IAEA hold, and allow the facility to segregate them (if desired) to make it easier for retrieval in the future. However, the MIS Working Group cautioned that EJ items other than these could be identified in the future. Decisions about EJ items are made each year based on results of surveillance and shelf-life studies. Since DE examinations for stress corrosion cracking of the ICCWR are now under way, new information from these examinations could alter the selection criteria for future EJ containers. While the MIS encouraged the facility to down blend S₁ containers to eliminate containers that have a higher potential for corrosion, MIS requested that the facility inform MIS of any S₁ containers being considered for down blending. In addition, MIS is working with the facility to develop guidance for collecting surveillance inspection information at the time of down blending (Section 7.)

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¹ Item H003032 was substituted for H003335, which was erroneously included in the May 13, 2020 letter to SRS,

[&]quot;3013 Container Selection for Destructive Examination." This will be corrected in the next letter.

Table 5-2. Twenty Potential Future EJ S₁ Containers for DE.

3013ContainerID	Reason Selected (All Percentages are wt%)
H003368	Best Moisture= 0.28% (DE performed on similar container - delay until near end of EJs to evaluate effects of aging)
H002560	Best Moisture =0.26% (Previously picked for FY 2019 but deferred to support operational constraints)
H003392	Best Moisture = 0.25%
H002778	Best Moisture =0.24% (DE performed on similar container - delay until near end of EJs to evaluate effects of aging)
H002557	Best Moisture = 0.24%
H003720	Best Moisture = 0.24%
H003670	Best Moisture = 0.23%
H003638	Best Moisture =0.21%, Cl = 8%, Mg = 2.4%, Weight gain = 0.6 g (high Cl and Mg and weight gain)
H004102	Best Moisture =0.19%, Weight gain = 1.9 g (high weight gain)
H003882 Best Moisture = 0.19%, Weight gain = 1.2 g, Am =0.31%, Cl%=5.9%, (High weight gain, differ material type, somewhat lower chloride and Am > 0.3%)	
H003729	Best Moisture = 0.19%, Re-Stabilized, Weight gain = 1.6 g (re-stabilized and high weight gain)
H003607 Best Moisture = 0.16%, CI = 2.5%, Mg = 1.4%, Weight gain = 1.1 g (CI/Mg ratio indicates N present which generates HCI)	
H002607	Best Moisture = 0.19%, CI = 5.4%, Mg = 1.7%, Weight gain = 1.3 g (Similar to H003607 with higher moisture)
H003828	Best Moisture = 0.21%, Am = 0.36%, Cl%=3.1%, (Am > 0.3%)
H003032	Best Moisture = 0.21%, Am = 0.31%, Cl%=11.5%, (Am > 0.3%)
H003830	Best Moisture = 0.18%, Chosen because re-stabilized and storage weight gain=0.5g
H002551	Best Moisture = 0.15%, Chosen because re-stabilized and storage weight gain = 0.2 g
H002802	Best Moisture = 0.13%, Chosen because re-stabilized and storage weight gain = 0.2 g
H003162	Best Moisture = 0.10%, Chosen because re-stabilized and storage weight gain = 0.2 g
Н003808	Best Moisture = 0.11%, Chosen because re-stabilized and storage weight gain = 0.1 g

Appendix A contains a list of S_1 containers remaining after 2022. The Appendix A list includes the future random sample containers, which also must be protected from down blending until after DE examination.

The 2015 ISP document directs that after the current random selection is complete in 2025, Field Surveillance should continue as long as containers continue to be stored. This will help address long-term aging effects and possible life extension, if necessary. In addition, it allows for surveillance of containers that continue to be generated (such as the ARIES containers described in Section 8.0).

6.0 DE ICCWR Examination Protocol to Meet the Confidence Criterion

An integral component of the 99.9% / 5% confidence criterion is that when one of the 61 randomly sampled containers is examined then (1) if a SCC crack exists in the ICCWR and is causing a leak of concern, there is an extremely high probability that it will be detected and (2) if there are conditions such that a crack of concern has a reasonable probability of occurring during the 50 year storage life of the container, then these conditions will be identified with extremely high probability. An ICCWR examination protocol has been developed "to ensure that if a container has a potential problem now or could have one in the future, it will be identified with extremely high probability during DE" (Martinez-Rodriguez and Kelly, 2021)

This protocol includes visual inspections, 35 mm pictures, stereo microscope images, helium leak testing, wet chemistry, Wide-Area 3D Measurement System (WAMS) imaging and analysis and laser confocal microscope (LCM) imaging and analysis. The imaging and analysis are followed by subsurface examination such as serial metallography, X-Ray Tomography (XRT) and plasma-Focused Ion Beam when needed to evaluate subsurface features and depths because some cracks may be longer under the surface than expected from the surface examination. Throughout the process the MIS-WG and the Corrosion Working Group (CWG), which consists of SMEs studying and assessing corrosion in 3013 containers, are consulted to determine if it is appropriate to proceed with the protocol or if additional imaging and analysis are needed.

7.0 SURVEILLANCE INSPECTION INFORMATION FROM DOWN BLENDING OPERATIONS

Down blending of material from 3013 containers provides an opportunity to collect valuable information about the 3013 container population. Down blending operations are described in Olson, 2020. The approach for collecting information at the time of down blending has not been finalized, however, the table below contains a proposed checklist for inspecting 3013 containers at the time of down blending.

Inside Outer Container	Corrosion (Y/N)	If Y, contact engineering
Outside of Inner container	Corrosion (Y/N)	If Y, contact engineering
Inner Container Lid	Corrosion as great or greater than worst-case lid (Y/N)	If Y, contact engineering
Inner Container Body	Coating, spots equal to or worse than Convenience Container examples (Y/N)	If Y, contact engineering
Cracks or holes in any containers	(Y/N)	If Y, contact engineering

Table 7-1. Inspection Checklist for 3013 Non-DE Containers*

A set of photographs to be used for training operators are currently being evaluated by down blending operators (Kelly, Veirs et al., 2021). These training photographs include an example of a worst-case lid to be reported, examples of lids that are not to be reported, examples of an inner

^{*}For non-DE's inspections may occur at any time.

container body that should be reported and an example of an inner container that does not need reporting. The photographs of the inner container needing reporting are actually of convenience containers, since an inner container with this level of corrosion has not been observed.

This down blending information will increase confidence that assumptions made to identify worst-case containers are correct. For example, an important worst-case assumption is that the containers from the P&C bin are considered to be bounding for corrosion. Another key assumption is that P&C containers having best moisture measurements ≥ 0.08 wt% (S₁) are much more likely to have significant corrosion than the remainder of the P&C population (S₂). Destructive examinations are focused on the S₁ population, which does not provide data to check these assumptions. However, down blending information is collected on all 3013 containers (S₁, S₂, Innocuous and Pressure bins), thereby providing information to check these assumptions. Assuming that no unexpected conditions are found, inspection information gained during down blending operations will also increase confidence that unexpected conditions have not been missed by insufficient sampling.

8.0 ARIES CONTAINERS ENTERING THE P&C POPULATION

The ARIES project at LANL has traditionally packaged 3013 containers with high-fired, high-purity plutonium oxide produced in the direct metal oxidation (DMO) process.

The ARIES population of 3013 outer containers is comprised of outer cans from several sources. The original DMO oxide production campaign, consisting of blend lots 1-5, is comprised of containers purchased from Washington Group under the original DOE contract. LANL 3013 containers designation A000xxx, consists of 44 containers that were packaged in 2009 through 2011 from LANL 00-1 materials and ARIES blend lots 1 through 5 (Table 8-1). These containers have been shipped to SRS, are in the PCD database and, therefore, are certified 3013s and in the ISP. Twenty-six of these containers are from LANL 00-1 material. Of these 26, 11 are in the Innocuous bin, 11 are in Pressure bin and four are in the P&C bin. Of the four P&C containers, one is in S₁, but it is not included in the random sample. The remaining 18 are packaged with traditional ARIES DMO oxide and are in the Innocuous bin.

Around 2011, 116 containers, originally purchased for Hanford and designated by H00XXXX were added to the LANL inventory (Table 8-1). This set comprises blend lots 16-54. These 116 are all traditional ARIES DMO oxide and are therefore innocuous. Thirty-five have been shipped to SRS, are in the PCD database and therefore in the ISP. They are all in the Innocuous bin. The remaining 81 are at LANL.

The final set of ARIES 3013 containers, starting with blend lot 55 (May 2017) are comprised of the containers purchased from Dynamic FlowForm (DFF). As of July 2021, these containers, designated by A002xxx, were used in the packaging of 114 containers, starting with blend lot 55. The use of the DFF containers for <u>innocuous</u> materials was authorized by DOE Savannah River in 2013, after evaluation of a separate suite of burst and drop tests. An additional 375 DFF outer containers remain unused in the LANL inventory of outer containers for use. At current production rates, LANL will utilize these containers through FY 2024, but is scheduled to

transition to packaging in the SAVY on a limited basis in FY 2023. All materials packaged in the DFF containers reside at LANL. As noted in the Introduction, currently only DFF containers with innocuous materials can be shipped to SRS.

Previously it was assumed that ARIES would generate five hundred 3013 containers and all of these would be in the Innocuous bin as part of the 2 Metric Tonne mission of record. This assumption was based on the ARIES planned production of containers for the mixed oxide (MOX) program, but with the termination of MOX, this plan was replaced with the dilute and dispose plan. Dilute and dispose is currently what constitutes all ARIES production. In addition to the termination of MOX, there have been a series of material redesignations within the National Nuclear Security Administration. These redesignations have introduced non-traditional materials to be processed under the ARIES umbrella. As of July 2021, ARIES has generated two hundred and seventy four (274) 3013 containers in total from the disposition of traditional ARIES material and other surplus-designated materials - 44 A000xxx (26 from 00-1 and 18 traditional ARIES DMO oxides), 116 H00xxxx (all traditional ARIES DMO oxides) and 114 A002xxx (traditional and non-traditional ARIES DMO oxides) (Table 8-1). Of the 114 A002xxx containers, all but nine (3 blend lots) are expected to meet the requirements for the Innocuous bin. These nine either have a history of possible Cl exposure or PG that indicates Cl, or have a Pu mass that is < 85 wt%, or both.

Table 8-1. Existing 274 ARIES containers

		At SRS	At LANL				
	Container	and in	not in				
Material	ID's	PCD	PCD	Total	Innocuous	Pressure	P&C
LANL 00-1 material	A000XXX	26	0	26	11	11	4
ARIES Blend lots 1 through 5							
(traditional ARIES DMO							
oxides)	A000XXX	18	0	18	18	0	0
ARIES Blend lots 16 through							
54 (traditional ARIES DMO							
oxides)	H00XXXX	35	81	116	116	0	0
ARIES Blend lots 55 and							
above (as of July 2021)							
(traditional and non-							
traditional ARIES DMO							
oxides)	A002XXX	0	114	114	105	0	9
Totals		79	195	274	250	11	13

To support the dilute and dispose efforts of SPD (surplus plutonium disposition), the ARIES program could generate up to 170 more 3013s by the end of 2023, before the transition to packaging in 2 QT SAVY containers. This would give a total of 444 ARIES generated 3013s.

The ARIES blending scheme has been tweaked to avoid generating P&C containers, nevertheless, it is estimated that in the worst-case situation there could be an additional 31 of the ARIES A002xxx containers in the P&C bin (including the nine mentioned above). This is in addition to the four 00-1 containers currently in the PCD database.

Subject to shipping and other logistical details, LANL may have to purchase additional 3013 containers depending on decisions by management regarding the implementation of packaging into the 2 QT SAVY. LANL plans to transition from 3013 packaging to the non-3013 compliant SAVY container when the SPD line item project completes CD-3. This decision point will greatly reduce LANL's risk of needing to store SAVY containers beyond their shelf life and associated risks for maintaining product specifications.

Currently LANL has plans for 3013s packaged with oxide to begin shipping in FY 2022. Specifically, LANL plans to ship 100 kg plutonium oxide, and in exchange, receive 100 kg of Alternate Feed Specification-2 metal for oxidation. The number of containers and other details have not been completed, although Shipper-Receiver documents are being developed to support both metal and oxide and are to be completed by the end of FY 2021.

Based on current ARIES plans and projections, at some point there could be approximately thirty-one certified ARIES 3013 containers in the P&C Bin that are at least five years old and were not considered in the random sampling. (The four currently in the PCD were in the ISP population at the time of random sampling.) It is not yet known how many of these 31 will be in S₁. The MIS will make a decision about surveillance of these ARIES containers when they are in the ISP and more information is available about moisture content and material composition.

TGA-MS data has been collected on the first 233 ARIES traditional DMO oxide containers (blend lots 1-78). The highest reported moisture is 0.04 wt%, and the highest packaging releative humidity is 7%. The chloride materials were packaged later and moisture results are not yet available. Current packaging continues at some risk while the TGA instrument is replaced, which is expected by the end of FY 2021.

9.0 SUMMARY

This 2021 Update documents changes to binning and surveillance sampling since 2016. Changes to the binning include moving twenty-six RFETS containers that were previously in the P&C higher moisture group (S₁) to S₂ based on revised best moisture measurements. This change resulted in the number containers required in the random sample to meet the confidence criterion decreasing from 62 to 61.

This Update also documents field surveillance activities from FY 2016 through FY 2021, provides the field surveillance plan for FY 2022 and documents plans for future sampling through FY 2025. Random sampling will be completed in FY 2025, if the plans described

in this Update are implemented. Field Surveillance in FY 2026 and later will be evaluated by the MIS-WG based on surveillance results, additional containers generated, and how quickly containers of potential concern are dispositioned.

Also included is a list of possible items for EJ in the future and a list of S_1 items remaining in the population after FY 2022, including the random sample items.

In addition, this Update describes a new effort to collect surveillance inspection information as part of down blending operations and describes ARIES 3013 containers entering the P&C population.

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APPENDIX A. LIST OF S₁ CONTAINERS REMAINING AFTER FY 2022

Table A-1 shows the remaining S_1 containers as of July 2021, excluding the containers proposed for DE in 2022. The first 15 containers are the remaining random selection containers identified for DE in FY 2023 and beyond. They are sorted by best moisture content, however, when they will be examined is not yet determined. The following S_1 containers in the table are sorted by 3013 Container ID.

 $Table \ \underline{A-1.\ S_1\ containers\ as\ of\ July\ 2021,\ excluding\ the\ containers\ proposed\ for\ DE\ in\ 2022}$

	3013 Container ID	DE Status	Best Moisture (%)
1	R601859	Future Random	0.27
2	H001314	Future Random	0.20
3	S002117	Future Random	0.14
4	H002631	Future Random	0.14
5	H002610	Future Random	0.14
6	S002203	Future Random	0.13
7	R600563	Future Random	0.13
8	H002766	Future Random	0.13
9	H004228	Future Random	0.12
10	H003639	Future Random	0.12
11	H004096	Future Random	0.12
12	H003469	Future Random	0.12
13	R610875	Future Random	0.09
14	H002798	Future Random	0.09
15	H002512	Future Random	0.08
16	A000570	Not yet Selected	0.20
17	H001221	Not yet Selected	0.26
18	H001223	Not yet Selected	0.29
19	H001282	Not yet Selected	0.13
20	H001289	Not yet Selected	0.29
21	H001327	Not yet Selected	0.26
22	H001344	Not yet Selected	0.16
23	H001404	Not yet Selected	0.15
24	H001421	Not yet Selected	0.18
25	H002426	Not yet Selected	0.19
26	H002462	Not yet Selected	0.15
27	H002465	Not yet Selected	0.13
28	H002468	Not yet Selected	0.23
29	H002474	Not yet Selected	0.08

Table A-1. S1 Containers (Continued)

	3013 Container ID	DE Status	Best Moisture (%)
20			
30	H002491	Not yet Selected	0.22
31	H002500	Not yet Selected	0.13
32	H002521	Not yet Selected	0.15
33	H002529	Not yet Selected	0.11
34	H002530	Not yet Selected	0.16
35	H002536	Not yet Selected	0.17
36	H002538	Not yet Selected	0.20
37	H002542	Not yet Selected	0.21
38	H002545	Not yet Selected	0.15
39	H002546	Not yet Selected	0.17
40	H002549	Not yet Selected	0.11
41	H002551	Not yet Selected	0.15
42	H002557	Not yet Selected	0.24
43	H002559	Not yet Selected	0.12
44	H002560	Not yet Selected	0.26
45	H002562	Not yet Selected	0.20
46	H002564	Not yet Selected	0.19
47	H002576	Not yet Selected	0.18
48	H002580	Not yet Selected	0.15
49	H002607	Not yet Selected	0.19
50	H002624	Not yet Selected	0.17
51	H002633	Not yet Selected	0.16
52	H002648	Not yet Selected	0.15
53	H002653	Not yet Selected	0.12
54	H002659	Not yet Selected	0.13
55	H002675	Not yet Selected	0.13
56	H002685	Not yet Selected	0.13
57	H002686	Not yet Selected	0.09
58	H002700	Not yet Selected	0.15
59	H002722	Not yet Selected	0.19
60	H002737	Not yet Selected	0.14
61	H002748	Not yet Selected	0.20
62	H002757	Not yet Selected	0.14
63	H002775	Not yet Selected	0.09
64	H002778	Not yet Selected	0.24
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Table A-1. S1 Containers (Continued)

	3013 Container ID	DE Status	Best Moisture (%)
65	H002782	Not yet Selected	0.14
66	H002785	Not yet Selected	0.12
67	H002802	Not yet Selected	0.13
68	H002806	Not yet Selected	0.15
69	H002809	Not yet Selected	0.10
70	H002836	Not yet Selected	0.08
71	H002913	Not yet Selected	0.11
72	H002982	Not yet Selected	0.11
73	H002989	Not yet Selected	0.19
74	H002990	Not yet Selected	0.11
75	H002997	Not yet Selected	0.09
76	H003032	Not yet Selected	0.21
77	H003042	Not yet Selected	0.14
78	H003080	Not yet Selected	0.10
79	H003113	Not yet Selected	0.08
80	H003162	Not yet Selected	0.10
81	H003175	Not yet Selected	0.15
82	H003249	Not yet Selected	0.15
83	H003260	Not yet Selected	0.09
84	H003303	Not yet Selected	0.15
85	H003309	Not yet Selected	0.12
86	H003312	Not yet Selected	0.08
87	H003313	Not yet Selected	0.12
88	H003318	Not yet Selected	0.12
89	H003319	Not yet Selected	0.11
90	H003323	Not yet Selected	0.12
91	H003325	Not yet Selected	0.10
92	H003326	Not yet Selected	0.12
93	H003330	Not yet Selected	0.10
94	H003334	Not yet Selected	0.08
95	H003335	Not yet Selected	0.23
96	H003336	Not yet Selected	0.15
97	H003340	Not yet Selected	0.17
98	H003353	Not yet Selected	0.10
99	H003355	Not yet Selected	0.11
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Table A-1. S1 Containers (Continued)

	3013 Container ID	DE Status	Best Moisture (%)
100	H003356	Not yet Selected	0.12
101	H003358	Not yet Selected	0.12
102	H003368	Not yet Selected	0.28
103	H003378	Not yet Selected	0.16
104	H003392	Not yet Selected	0.25
105	H003410	Not yet Selected	0.11
106	H003411	Not yet Selected	0.17
107	H003413	Not yet Selected	0.10
108	H003445	Not yet Selected	0.14
109	H003468	Not yet Selected	0.11
110	H003489	Not yet Selected	0.12
111	H003499	Not yet Selected	0.17
112	H003515	Not yet Selected	0.15
113	H003569	Not yet Selected	0.12
114	H003574	Not yet Selected	0.20
115	H003579	Not yet Selected	0.09
116	H003586	Not yet Selected	0.16
117	H003588	Not yet Selected	0.16
118	H003597	Not yet Selected	0.12
119	H003598	Not yet Selected	0.17
120	H003599	Not yet Selected	0.19
121	H003600	Not yet Selected	0.19
122	H003604	Not yet Selected	0.16
123	H003607	Not yet Selected	0.16
124	H003613	Not yet Selected	0.21
125	H003615	Not yet Selected	0.21
126	H003622	Not yet Selected	0.16
127	H003635	Not yet Selected	0.20
128	H003638	Not yet Selected	0.21
129	H003642	Not yet Selected	0.13
130	H003643	Not yet Selected	0.14
131	H003652	Not yet Selected	0.15
132	H003664	Not yet Selected	0.13
133	H003666	Not yet Selected	0.15
134	H003667	Not yet Selected	0.15

Table A-1. S₁ Containers (Continued)

	3013 Container ID	DE Status	Best Moisture (%)
135	H003669	Not yet Selected	0.18
136	H003670	Not yet Selected	0.23
137	H003671	Not yet Selected	0.08
138	H003672	Not yet Selected	0.17
139	H003679	Not yet Selected	0.18
140	H003683	Not yet Selected	0.19
141	H003687	Not yet Selected	0.13
142	H003690	Not yet Selected	0.17
143	H003700	Not yet Selected	0.18
144	H003701	Not yet Selected	0.18
145	H003705	Not yet Selected	0.15
146	H003711	Not yet Selected	0.14
147	H003716	Not yet Selected	0.20
148	H003718	Not yet Selected	0.11
149	H003720	Not yet Selected	0.24
150	H003727	Not yet Selected	0.14
151	H003729	Not yet Selected	0.19
152	H003738	Not yet Selected	0.17
153	H003749	Not yet Selected	0.12
154	H003757	Not yet Selected	0.13
155	H003767	Not yet Selected	0.08
156	H003772	Not yet Selected	0.08
157	H003782	Not yet Selected	0.13
158	H003802	Not yet Selected	0.14
159	H003806	Not yet Selected	0.12
160	H003808	Not yet Selected	0.11
161	H003816	Not yet Selected	0.09
162	H003817	Not yet Selected	0.11
163	H003828	Not yet Selected	0.21
164	H003829	Not yet Selected	0.14
165	H003830	Not yet Selected	0.18
166	H003837	Not yet Selected	0.11
167	H003847	Not yet Selected	0.17
168	H003855	Not yet Selected	0.10
169	H003864	Not yet Selected	0.12

Table A-1. S₁ Containers (Continued)

Table A-1. Si Containers (Continued)				
	3013 Container ID	DE Status	Best Moisture (%)	
170	H003870	Not yet Selected	0.12	
171	H003871	Not yet Selected	0.17	
172	H003878	Not yet Selected	0.10	
173	H003882	Not yet Selected	0.19	
174	H003892	Not yet Selected	0.11	
175	H003895	Not yet Selected	0.22	
176	H003910	Not yet Selected	0.12	
177	H003913	Not yet Selected	0.09	
178	H003916	Not yet Selected	0.17	
179	H003918	Not yet Selected	0.13	
180	H003930	Not yet Selected	0.11	
181	H003940	Not yet Selected	0.16	
182	H003944	Not yet Selected	0.20	
183	H003948	Not yet Selected	0.12	
184	H003953	Not yet Selected	0.15	
185	H003959	Not yet Selected	0.13	
186	H003968	Not yet Selected	0.16	
187	H003970	Not yet Selected	0.14	
188	H003996	Not yet Selected	0.11	
189	H003998	Not yet Selected	0.09	
190	H004000	Not yet Selected	0.09	
191	H004014	Not yet Selected	0.10	
192	H004015	Not yet Selected	0.10	
193	H004017	Not yet Selected	0.10	
194	H004027	Not yet Selected	0.13	
195	H004029	Not yet Selected	0.17	
196	H004031	Not yet Selected	0.09	
197	H004036	Not yet Selected	0.16	
198	H004046	Not yet Selected	0.11	
199	H004047	Not yet Selected	0.10	
200	H004051	Not yet Selected	0.10	
201	H004052	Not yet Selected	0.15	
202	H004053	Not yet Selected	0.19	
203	H004054	Not yet Selected	0.21	
204	H004064	Not yet Selected	0.10	

Table A-1. S1 Containers (Continued)

	3013 Container ID	DE Status	Best Moisture (%)
205	H004066	Not yet Selected	0.14
206	H004069	Not yet Selected	0.12
207	H004071	Not yet Selected	0.15
207	H004072	Not yet Selected	0.08
	H004073	Not yet Selected	0.09
209		Not yet Selected	0.09
210	H004079	•	
211	H004083	Not yet Selected	0.21
212	H004084	Not yet Selected	0.09
213	H004093	Not yet Selected	0.12
214	H004098	Not yet Selected	0.10
215	H004100	Not yet Selected	0.13
216	H004102	Not yet Selected	0.19
217	H004104	Not yet Selected	0.12
218	H004105	Not yet Selected	0.12
219	H004106	Not yet Selected	0.13
220	H004114	Not yet Selected	0.13
221	H004115	Not yet Selected	0.08
222	H004117	Not yet Selected	0.16
223	H004119	Not yet Selected	0.17
224	H004122	Not yet Selected	0.18
225	H004125	Not yet Selected	0.12
226	H004127	Not yet Selected	0.11
227	H004128	Not yet Selected	0.12
228	H004137	Not yet Selected	0.10
229	H004151	Not yet Selected	0.12
230	H004152	Not yet Selected	0.10
231	H004157	Not yet Selected	0.14
232	H004158	Not yet Selected	0.09
233	H004160	Not yet Selected	0.11
234	H004161	Not yet Selected	0.18
235	H004163	Not yet Selected	0.11
236	H004164	Not yet Selected	0.09
237	H004165	Not yet Selected	0.17
238	H004166	Not yet Selected	0.10
239	H004168	Not yet Selected	0.08
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Table A-1. S1 Containers (Continued)

	3013 Container ID	DE Status	Best Moisture (%)
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240	H004169	Not yet Selected	0.14
241	H004176	Not yet Selected	0.10
242	H004180	Not yet Selected	0.08
243	H004182	Not yet Selected	0.12
244	H004185	Not yet Selected	0.14
245	H004189	Not yet Selected	0.10
246	H004194	Not yet Selected	0.13
247	H004203	Not yet Selected	0.16
248	H004204	Not yet Selected	0.19
249	H004207	Not yet Selected	0.11
250	H004223	Not yet Selected	0.10
251	H004231	Not yet Selected	0.13
252	H004232	Not yet Selected	0.10
253	H004233	Not yet Selected	0.10
254	H004236	Not yet Selected	0.09
255	H004237	Not yet Selected	0.09
256	H004238	Not yet Selected	0.12
257	H004239	Not yet Selected	0.13
258	H004240	Not yet Selected	0.12
259	H004241	Not yet Selected	0.15
260	H004242	Not yet Selected	0.15
261	H004246	Not yet Selected	0.11
262	H004248	Not yet Selected	0.10
263	H004249	Not yet Selected	0.14
264	H004250	Not yet Selected	0.15
265	H004252	Not yet Selected	0.18
266	H004254	Not yet Selected	0.14
267	H004271	Not yet Selected	0.11
268	H004315	Not yet Selected	0.13
269	H004392	Not yet Selected	0.09
270	R600151	Not yet Selected	0.26
271	R600310	Not yet Selected	0.10
272	R600511	Not yet Selected	0.13
273	R600546	Not yet Selected	0.22
274	R600557	Not yet Selected	0.10
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Table A-1. S1 Containers (Continued)

	3013 Container ID	DE Status	Best Moisture (%)
275	R600582	Not yet Selected	0.11
276	R600599	Not yet Selected	0.09
277	R600627	Not yet Selected	0.13
278	R600696	Not yet Selected	0.15
279	R600722	Not yet Selected	0.25
280	R600862	Not yet Selected	0.15
281	R600956	Not yet Selected	0.23
282	R601023	Not yet Selected	0.10
283	R601048	Not yet Selected	0.17
284	R601353	Not yet Selected	0.12
285	R601561	Not yet Selected	0.20
286	R601724	Not yet Selected	0.08
287	R601774	Not yet Selected	0.18
288	R601861	Not yet Selected	0.09
289	R601875	Not yet Selected	0.20
290	R601876	Not yet Selected	0.10
291	R601929	Not yet Selected	0.12
292	R602007	Not yet Selected	0.10
293	R602012	Not yet Selected	0.20
294	R602533	Not yet Selected	0.10
295	R602586	Not yet Selected	0.13
296	R602787	Not yet Selected	0.17
297	R610563	Not yet Selected	0.14
298	R610644	Not yet Selected	0.12
299	R610682	Not yet Selected	0.09
300	R610747	Not yet Selected	0.10
301	R610751	Not yet Selected	0.21
302	R610758	Not yet Selected	0.10
303	R610770	Not yet Selected	0.09
304	R610856	Not yet Selected	0.08
305	R611073	Not yet Selected	0.17
306	R611271	Not yet Selected	0.09
307	R611307	Not yet Selected	0.09
308	S000730	Not yet Selected	0.08
309	S000867	Not yet Selected	0.08
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Table A-1. S1 Containers (Continued)

Table A-1. 51 Containers (Continueu)			
	3013 Container ID	DE Status	Best Moisture (%)
310	S002105	Not yet Selected	0.13
311	S002110	Not yet Selected	0.12
312	S002113	Not yet Selected	0.09
313	S002118	Not yet Selected	0.17
314	S002130	Not yet Selected	0.14
315	S002135	Not yet Selected	0.13
316	S002136	Not yet Selected	0.12
317	S002139	Not yet Selected	0.20
318	S002148	Not yet Selected	0.21
319	S002152	Not yet Selected	0.14
320	S002181	Not yet Selected	0.15
321	S002187	Not yet Selected	0.21
322	S002212	Not yet Selected	0.16
323	S002221	Not yet Selected	0.16
324	S002222	Not yet Selected	0.08
325	S002251	Not yet Selected	0.09
326	S002271	Not yet Selected	0.10
327	S002275	Not yet Selected	0.08
328	S002284	Not yet Selected	0.11
329	S002286	Not yet Selected	0.12